1272230

CB&I
2700 Chandler Avenue, Building C
Las Vegas, NV 89120
Tel: +1 702 795 0515
Fax: +1 702 795 8210
www.CBI.com

November 14, 2013

Dania Zinner
USEPA; Region 8
1595 Wynkoop Street (8EPR-SR)
Denver, CO 80202-1129

Document ID #: 2019-11142013-2

Dear Ms. Zinner:

EPA CONTRACT NUMBER EP-W-10-033
TASK ORDER NUMBER 2019
QA SUPPORT FOR THE LIBBY ASBESTOS SITE

Enclosed please find the Summary Asbestos On-site Audit Report for the on-site audit performed on July 9, 2013 at EMSL Analytical, Inc. in Libby, Montana. This report and the accompanying checklist are deliverables under Task 5 of the subject Task Order.

If you have any questions, please feel free to contact me.

Sincerely,

Timothy L. Vonnahme
Audit Group Manager, QATS Program
CB&I Federal Services, LLC
Phone: (702) 895-8729
E-Mail Address: timothy.vonnahme@cbifederalservices.com

cc: Administrative Contracting Officer (letter only)
Audit Group Files

REPORT

FOR

TASK ORDER NUMBER 2019
QUALITY ASSURANCE SUPPORT FOR THE LIBBY ASBESTOS SITE

SUMMARY ASBESTOS ON-SITE AUDIT REPORT

EMSL Analytical, Inc. (Libby, MT)

Prepared by:

The Data Auditing Group
Quality Assurance Technical Support Program
CB&I Federal Services, LLC
2700 Chandler Avenue
Las Vegas, Nevada 89120

November 14, 2013

QATS Contract Number: EP-W-10-033

Prepared for:

Dania Zinner
Task Order Manager

Region 8
U.S. Environmental Protection Agency
1595 Wynkoop Street
Denver, CO 80202

TABLE OF CONTENTS

ATTACHMENT

LABORATORY INFORMATION AND AUDIT SCOPE

This report summarizes the results of an asbestos on-site laboratory audit of EMSL Analytical, Inc. in Libby, Montana performed on July 9, 2013. The audit was conducted in support of United States Environmental Protection Agency (EPA) Region 8 Libby Superfund Site activities. The purpose of the audit was to evaluate corrective actions taken by the laboratory to address deficiencies identified from the last on-site audit conducted on August 8-9, 2012, and to investigate analytical inconsistencies detailed in a technical memo provided to EPA on September 20, 2013, (**see Attachment 2**). CB&I Federal Services, LLC Quality Assurance Technical Support (QATS) staff participation in the on-site audit and subsequent preparation of this report was performed under Task 5, Task Order 2019, of QATS Contract EP-W-10-033.

Detailed information regarding the subject laboratory is as follows:

Date of On-site:	**July 9, 2013**
Laboratory:	**EMSL Analytical, Inc.** **107 West 4th Street** **Libby, Montana 59923** **406.293.9066**
Special Projects Manager:	**Robyn Denton**
Audit Team	
US EPA:	**Christina Progess, Remedial Project Manager, Superfund, Region 8**
CB&I QATS:	**Michael P. Lenkauskas, CQA, Lead Auditor**

The Audit Team, comprised of EPA Region 8 and CB&I Federal Services, LLC QATS personnel, performed the technical and evidentiary aspects of the on-site audit. Both the technical and evidentiary parts of the audit involved an evaluation of corrective actions taken by the laboratory to address the deficiencies identified during the previous on-site audit conducted on August 8-9, 2012, and also evaluation of those laboratory areas and data associated with the issues described in the technical memo submitted to EPA on September 20, 2013.

The processes evaluated included sample receipt, storage, tracking, direct and indirect sample preparation for Transmission Electron Microscopy (TEM) analysis, analysis by Polarized Light Microscopy (PLM), and analysis by TEM. All pertinent laboratory instrumentation and equipment were inspected for proper maintenance and calibration, and laboratory personnel were interviewed to determine their understanding and adherence to laboratory procedures.

During the course of the audit, the applicable sections of the Libby-Specific Asbestos Laboratory On-site Audit Checklist were completed by the QATS Audit Team. Sections of the checklists not completed during the audit are indicated with an “NA.” The checklist is provided as an attachment to this report (EPA only).

EXECUTIVE SUMMARY

An asbestos on-site audit was performed at the EMSL Analytical, Inc. in Libby, Montana on July 9, 2013 in support of EPA Region 8 Libby Superfund Site activities. The primary focus of the audit was to evaluate the corrective actions taken by the laboratory to address the deficiencies identified during the previous on-site audit conducted on August 8-9, 2012, and also those areas and data associated with the issues described in the technical memo submitted to EPA on September 20, 2013. The laboratory areas and processes evaluated included sample receipt, storage, tracking, direct and indirect preparation of samples for TEM analysis, analysis by PLM, and analysis by TEM.

The corrective actions applied by the laboratory to the nine deficiencies identified in the August 2012 on-site audit were evaluated during the current on-site audit. The Audit Team determined that the laboratory had completely addressed all nine deficiencies, for a corrective action rate of 100%.

The on-site audit identified five new deficiencies which are summarized by laboratory area below:

Sample Receipt, Storage, Log-in, and Chain-of-Custody (COC) – One deficiency was assessed for water samples stored in the basement sample storage area that did not exhibit a laboratory sample identification number.

Indirect and Direct Preparation of Air Filter and Dust Samples – Four deficiencies were assessed: the pan balance, to be used by the laboratory for the gravimetric analyses, has not been calibrated by a certified technician; a logbook to record calibration of the pan balance has not been assigned; the hood used for treatment of water samples by Ozone and UV light does not have a brim to contain liquids in the hood; and the ashing furnace has not been calibrated and does not have an assigned calibration logbook.

Special Investigation – EPA directed QATS to investigate the following four issues associated with the analysis of Operable Unit 3 (OU3) samples: TEM Inter-lab sample preparation issues; inadequate frequency of project-specific QC analyses; possible misidentification of samples; and result discrepancies between TEM rapid turn-around-time (TAT) and full analysis of OU3 water samples. The laboratory addressed the Inter-lab sample preparation issues and inadequate frequency of project-specific QC analyses prior to the on-site evaluation (**see Attachment 2**). The misidentification of samples was thought to be associated with the failure to assign laboratory identification numbers to all samples (see Comment 1); however, this root cause was not conclusively determined. The source of the result discrepancies between TEM rapid TAT and full analysis of OU3 water samples was not determined by the Audit Team during the audit; however, following the audit, it was determined that the TEM analysts were not performing the required elemental or the diffraction pattern analyses necessary to identify structures as asbestos. Instead they were incorrectly reporting diatom fragments as countable asbestos structures (i.e. LA, WRTA, NaK).

In addition to adequately addressing the deficiencies identified in the previous on-site audit, assisting the Audit Team in their investigation of the issues detailed in the technical memo to EPA dated September 20, 2013. All staff and management were cooperative, readily answered all questions asked by the Audit Team, and appeared to be responsive to the identified deficiencies.

AUDIT DEFICIENCIES

Sample Receipt, Storage, Log-in, and Chain-of-Custody (COC)

At the time of this audit, the laboratory had recently expanded into adjoining office space within the building, which includes a new reception area used to both accept visitors and receive samples. The Audit Team found this area to be clean and well organized. The evaluation of this area focused on two deficiencies identified in the previous audit associated with sample login record keeping and sample storage. Both of these deficiencies were found to have been addressed as described in the section "Corrective Action Applied from the Previous Audit Deficiencies" on page 7 of this report. While evaluating the sample storage area, one new deficiency was identified:

1. Water samples stored in the basement sample storage area do not exhibit laboratory sample identification numbers. The requirement that laboratory order numbers be physically attached to each sample batch is described in Section 5.8.4 of the Laboratory's Quality Assurance manual (QAM). (Checklist No. 4.4.4)

   Recommended Corrective Action – In order to ensure samples are properly tracked and identified, all samples must exhibit a unique laboratory sample identification number. The samples already in storage should be labeled retroactively.

Indirect and Direct Preparation of Air Filter and Dust Samples

The sample preparation area has also been recently expanded, offering more space to perform the current sample preparation techniques, but also to provide laboratory capabilities to eventually prepare duff and bark samples. The evaluation of this area focused on deficiencies from the previous on-site audit and on new equipment added as a result of the expansion. A previous deficiency related to obsolete calibration documentation was found to have been addressed. Four (4) new deficiencies were identified in this area, all related to facility expansion:

2. The hood where water samples are treated by Ozone and UV light does not have a brim or other form of containment to prevent spilled liquids from exiting the hood and spilling into the general laboratory area. The requirement that contamination of samples, the laboratory environment, and reagents used in analysis be avoided in order to provide the highest quality, legally defensible data is described in Section 5.3.2 of the Laboratory's QAM. (Checklist No. 6.3)

   Recommended Corrective Action – Ensure that the proper engineering controls are in place to both protect laboratory personnel from exposure and minimize the potential for laboratory contamination.

3. The newly acquired furnace that will be used for the ashing of tree bark, duff, and other samples, has not been calibrated and does not have an associated logbook to document calibration. The requirement that a logbook be maintained for each piece of critical equipment in use at the laboratory to record all maintenance, repairs, calibrations performed is described in Section 5.5.1 of the Laboratory's QAM. (Checklist No. 6.4.3.1)

   Recommended Corrective Action – Ensure that logbooks are maintained for each piece of critical equipment in use at the laboratory to record all maintenance, repairs, calibrations performed.

4. The recently acquired pan balance that will be used for the gravimetric analyses associated with the preparation of tree bark, duff, and other samples, does not have a logbook for recording calibration results. The requirement that a logbook be maintained for each piece of critical equipment in use at the laboratory to record all maintenance, repairs, calibrations performed is described in Section 5.5.1 of the Laboratory's QAM. (Checklist No. 6.4.4.1)

   Recommended Corrective Action – Ensure that logbooks are maintained for each piece of critical equipment in use at the laboratory to record all maintenance, repairs, and calibrations performed.

5. The recently acquired pan balance that will be used for the gravimetric analyses associated with the preparation of tree bark, duff, and other samples, has not been calibrated by a certified technician. The requirement that balances be calibrated upon installation and then annually thereafter by an outside accredited calibration provider is described in Section 5.5.3.1 of the Laboratory's QAM. (Checklist No. 6.4.4.2)

   Recommended Corrective Action – Ensure that all balances are calibrated upon installation and annually thereafter by an outside accredited calibration provider.

Transmission Electron Microscopy (TEM) Analysis

The evaluation of this area focused on a deficiency from the previous on-site audit related to intra-laboratory analysis tracking, which was found to have been adequately addressed. There were no new deficiencies observed.

Polarized Light Microscopy (PLM) Analysis

The evaluation of this area focused on deficiencies from the previous on-site audit related to reference slide management and use, which were found to have been adequately addressed. There were no new deficiencies observed.

Data Management

This area was not evaluated since there was no data management issues identified in the August 2012 audit.

Quality Control and Quality Assurance (QA/QC)

This area was not evaluated since there was no QA/QC issues identified in the August 2012 audit.

Special Investigation

On June 4, 2013 a memorandum prepared by QATS and CDM Smith detailed a number of issues and analytical discrepancies that had recently been observed in data from the EMSL Libby laboratory. This memo detailed the following issues associated with the analysis of Operable Unit 3 (OU3) samples:

- TEM Inter-lab sample preparation issues
- Inadequate frequency of project-specific QC analyses
- Possible misidentification of samples
- Result discrepancies between TEM rapid TAT and full analysis of OU3 water samples.

As part of the on-site audit, Region 8 tasked QATS whether the deficiencies had been corrected. EPA Region 8 forwarded this memo to the laboratory and requested corrective action to be performed. The inter-lab sample preparation and inadequate QC analysis issues were found to have been resolved prior to the on-site audit, and are described in detail in the attached updated version of this memorandum. Concerning the possible misidentification of samples, the Audit Team thought this could possibly be attributed the laboratory not exhibiting the unique laboratory identifier on samples upon receipt (refer to Comment #1); however, this relationship was determined to be inconclusive. To investigate the result discrepancies between the rapid TAT and full TEM analysis of samples that were analyzed by both methods, the Audit Team interviewed involved personnel, reviewed the associated data, and looked at the associated sample containers, filter preparations, and TEM grids, but could not identify the source of the result discrepancies. Although this issue was not resolved during the on-site audit, it was later determined that the TEM analysts were not performing the required elemental or the diffraction pattern analyses necessary to identify structures as asbestos, but instead were incorrectly reporting diatom fragments as countable asbestos structures (i.e. LA, WRTA, NaK). The results of these and the other findings are described in the most recent revision of the memorandum, which is provided as an attachment to this report (**see Attachment 2**).

CORRECTIVE ACTION APPLIED FROM THE PREVIOUS AUDIT DEFICIENCIES

The on-site laboratory evaluation included an assessment of the nine (9) deficiencies reported in the previous Summary Asbestos On-site Audit Report for the on-site audit performed on August 8-9, 2012. The Audit Team determined that the laboratory had completely addressed all nine deficiencies resulting in a correction rate of 100%. The following are the deficiencies identified from the previous on-site audit, the laboratory's verbatim responses to the deficiencies, and the effectiveness checks made during the current on-site audit.

Sample Receipt, Storage, Log-in, and Chain-of-Custody (COC)

1. The EPA Region 8 Libby Site Investigation Logbook, which is used to record the transfer of samples, prepared samples, hard copy deliverables, and electronic deliverables to and from the laboratory does not include a field describing what is being transferred (i.e. data or samples). The requirement that data are recorded and identifiable to the task is described in Section 4.13.2 of the laboratory's QAM. (Checklist Nos. 4.7.1, 9.2.1 and 9.2.2)

   Recommended Corrective Action – Ensure that data entries are identifiable to the task described.

   Laboratory Response (10/10/2012): *Immediately upon being pointed out to us, the policy was changed to include what (sample or data) was being sent or received (see attached).*

   Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

2. Water samples waiting to be prepared and analyzed for asbestos by TEM are stored on the floor in the basement. The requirement that samples be stored in a manner which provides protection from possible contamination or loss of integrity is described in Section 5.8.5 of the laboratory's QAM. (Checklist No. 4.4.1)

Recommended Corrective Action – Ensure that samples are stored in manner which minimizes the possibility of contamination or loss of integrity.

Laboratory Response (10/10/2012*): Shelving was cleaned out and the samples moved off of the floor.*

Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

Indirect and Direct Preparation of Air Filter and Dust Samples

3. The calibrated time required to perform the filter etching procedure posted on the instrument was not the same time as determined from the most recent quarterly calibration, but from a calibration performed in 2007. The requirement that obsolete documents be removed from the laboratory or, if they are to be maintained for historical reference, isolated so that they are not accidentally used is described in Section 4.3.1.4 of the laboratory's QAM. (Checklist No. 6.4.5.1)

   Recommended Corrective Action – Ensure that obsolete documents are either removed from the laboratory or archived to prevent their use.

   Laboratory Response (10/10/2012): *A new sticker has been placed on the asher and in the future the result of the most recent calibration will be attached to the asher in addition to being programmed into the timer (see attached memo). .*

   Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

Transmission Electron Microscopy (TEM) Analysis

4. The TEM QC Logbook used to track and ensure that intra-laboratory analyses (i.e. recount same [RS] and recount different [RD]) are performed at the correct frequency is not completed in a timely manner. The column used to record the identification of the client sample used for Quality Control (QC) analysis had not been completed for many of the QC analyses performed from 9/11/2011 to present. The requirement to record analyses at the time they are performed is described in Section 4.13.2 of the laboratory's QAM. (Checklist No. 7.15.1)

   Recommended Corrective Action – Ensure that entries to logbooks and other preprinted documents are made in a timely manner.

   Laboratory Response (10/10/2012): *Sample numbers are now recorded into the QC logbook as soon as the sample is identified per lab mod LB000029b (see attached memo). Analysis results are entered immediately upon completion of analysis.*

   Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

Polarized Light Microscopy (PLM) Analysis

5. The PLM analyst, and not the QC Coordinator, currently maintain and manage the reference slides used to monitor analyst accuracy. This could result in the analyst becoming familiar with the true values of the reference slides. The requirement that the

laboratory's QA program be implemented and managed by the QA coordinator is described in Section 5.9.1 of the laboratory's QAM. (Checklist No. 8.13.1)

Recommended Corrective Action – Ensure that QC reference materials are stored and managed in a manner that ensures their true values remain unknown.

Laboratory Response (10/10/2012): *Control of the true value key for the PLM reference set has been transferred to the QA manager.*

Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

6. A set of laboratory prepared and permanently mounted LA reference slides of 0.2% and 1.0% are not available for use in the qualitative determination of LA in fine ground soil samples. The requirement that laboratories analyzing samples for LA prepare five slide-mounts from the 0.2% and 1.0% LA reference materials in a permanent medium, such as epoxy or melt-mount, is described in Section 13.7.3.4 of the Libby-specific SOP for the Analysis of Fibers in Soil by PLM (SRC-Libby-03, Rev. 3). (Checklist No. 8.11.6.3)

   Recommended Corrective Action – Ensure that a permanent set of laboratory-specific slide-mounts of the 0.2% and 1.0% LA are available to assist in the semi-quantitative estimation of LA in fine ground soil samples.

   Laboratory Response (10/10/2012): *Slides of the 0.2% and the1.0% reference material have been made.*

   Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

Data Management

No observations concerning data management were identified.

Quality Control and Quality Assurance

7. Internal audits, which are scheduled to be performed annually, have not been performed since January 2011. In addition, the checklist completed for the most recent internal audit did not include documentation of when it was performed, where it was performed, or who it was performed by. The requirements for performing and documenting annual internal audits are described in Section 4.14 of the laboratory's QAM. (Checklist No. 10.3.1)

   Recommended Corrective Action – Ensure that internal audits are properly recorded and performed on an annual basis.

   Laboratory Response (10/10/2012): *The 2012 internal audit had not been done at the time of the audit. The internal audit for the calendar year 2012 has been performed. It was done 24-25 September 2012.*

   Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

8. Corrective and preventive actions are initiated and tracked using an outdated system. Corrective and preventive actions are recorded on obsolete pre-printed documents and not the electronic corrective action forms described in the laboratory's written procedures. The requirements for documenting and tracking corrective and preventive actions are described in Section 4.11 of the laboratory's QAM and SOP for non-conformities and corrective actions. (Checklist No. 10.4.1)

   Recommended Corrective Action – Ensure that the laboratory's current system for documenting and tracking corrective actions is utilized.

   Laboratory Response (10/10/2012): *The pre-printed forms were used only to record orthographic errors and the issuance of the revised reports resulting. This project, with the extensive data validation, is such that something as trivial as a one minute variance in the time received would necessitate the issue of a revised report. The electronic form is used for substantive issues such as response to this audit.*

   Revised Laboratory Response (12/18/2012): *The laboratory is now using the corrective action procedures following the quality system program documented in our SOP. All non-conformities are recorded, evaluated and tracked using the corrective action workbook. The use of pre-printed forms and hand written entries have been discontinued.*

   Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

9. While performing the on-site audit, an e-mail was received from ESAT Region 8 notifying the Audit Team of contamination detected in a sample collected within the laboratory on 7/20/2012. However, there was no evidence that the required corrective actions including cleaning and resampling had been initiated by the laboratory. When questioned concerning this issue the laboratory manager stated they had been informed and that the laboratory had cleaned the area, but that another sample had not been collected and a corrective action had not been initiated. The requirements to initiate a corrective action report, clean the effected area, and collect additional samples to ensure the area is free of contamination is described in Section A.5.3.2.2 of the laboratory's QAM. (Checklist No. 10.6.2.2)

   Recommended Corrective Action – In the event that contamination is detected during quarterly ambient air monitoring, ensure that a corrective action is initiated. This includes cleaning the area and collecting additional ambient air samples to document the area is free of contamination.

   Laboratory Response (10/10/2012): *.We were informed on 7 August 2012 that a single fiber had been detected in a sample collected in the PLM room during the monthly air monitoring 20 July 2012. The PLM room was wiped down, HEPA vacuumed, and the hood HEPA filter replaced 7 August 2012. A re-preparation of the original filter proved to be ND. A duplicate sample taken at the same time was also ND. In discussions with the ESAT technical consultant, it was his opinion that the single fiber was a random event as is sometimes encountered in the ambient air samples collected in Libby. In his opinion, no further action was necessary. Another air sample was taken the afternoon of 9 August 2012 that was also ND. Another sample taken in September was also ND.*

Revised Laboratory Response (12/18/2012): *Going forward the laboratory will ensure that re-sampling will be conducted immediately following the clean up procedure as documented in our SOP.*

Effectiveness Check (07/09/2013): This finding was found to have been adequately addressed.

CONCLUSIONS

An asbestos laboratory on-site audit of EMSL Analytical, Inc. in Libby, Montana was performed on July 9, 2013 in support of EPA Region 8 Libby Superfund Site activities. The primary focus of the audit involved an evaluation of corrective actions taken by the laboratory to address the deficiencies identified during the previous on-site audit conducted on August 8-9, 2012. The laboratory areas and process evaluated include sample receipt, sample storage, sample tracking, direct and indirect sample preparation for Transmission Electron Microscopy (TEM) analysis, analysis by TEM, and analysis by Polarized Light Microscopy (PLM).

The Audit Team evaluated the corrective action applied to the nine deficiencies identified in the previous on-site audit and determined that the laboratory completely addressed all nine deficiencies, for a corrective action rate of 100%.

The on-site audit identified the following five new deficiencies:

- Water samples stored in the basement samples storage area do not exhibit a laboratory sample number.
- No laboratory logbook is assigned to record calibration activities for the pan balance.
- The laboratory pan balance has not been calibrated by a certified technician.
- The hood where water samples are treated by Ozone and UV light does not have a brim or other form of containment to prevent spilled liquids from exiting the hood.
- The furnace has not been calibrated to the appropriate temperatures, nor has an associated calibration log been developed.

EPA directed QATS to investigate as part of the on-site audit four recently identified issues associated with data from the analysis of Operable Unit 3 (OU3) samples. These include TEM Inter-lab sample preparation issues; inadequate frequency of project-specific QC analyses; possible misidentification of samples; and result discrepancies between TEM rapid TAT and full analysis of OU3 water samples. The laboratory addressed the Inter-lab sample preparation issues and inadequate frequency of project-specific QC analyses prior to the on-site evaluation (**see Attachment 2**). The misidentification of samples was thought to be associated with failure to assign laboratory identification numbers to all samples (see Comment 1); however, this root cause was not conclusively determined. The Audit Team did not determine during the audit the source of the result discrepancies between TEM rapid TAT and full analysis of OU3 water samples; however, following the audit it was determined the TEM analysts were not performing the required elemental or the diffraction pattern analyses necessary to identify structures as asbestos, but instead were incorrectly reporting diatom fragments as countable asbestos structures (i.e. LA, WRTA, NaK).

With the exception of the identified deficiencies, the on-site evaluation revealed the laboratory to have sufficient facilities, equipment, and staff to effectively analyze samples in accordance with the specified methodologies and Libby-specific protocol. All staff and management were cooperative, readily answered all questions asked by the Audit Team, and appeared to be responsive to the identified audit deficiencies.

ATTACHMENT 1

Libby-Specific Asbestos Laboratory On-site Audit Checklist (EPA Only)

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site: 7/9/2013

Laboratory: EMSL Analytical, Inc.

Address: 107 West 4th Street

Libby, Montana 59923

Telephone: (406) 293-9066

Laboratory Personnel Contacted

Name	Title
Roy Pescador	Laboratory Manager
Elisabeth JoMay Wyatt Pescador	Assistant Laboratory Manager
Deven Barney	TEM Sample Preparation
Kelly Colberg	PLM Analyst
Margi Carr	PLM Analyst

Evaluation Team

Name	Title
Christina Progess	USEPA Superfund Project Manager
Michael Lenkauskas, CQA	CB&I Federal Services, LLC (QATS), Senior Auditor

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

1.0 LABORATORY STATUS & CAPABILITIES	Yes	No	Comments
1.1 Which of the following capabilities does the laboratory possess:			
1.1.1 Phase Contrast Microscopy (PCM)?	☒	☐	
1.1.2 Polarized Light Microscopy (PLM)?	☒	☐	
1.1.3 Transmission Electron Microscopy (TEM)?	☒	☐	
1.1.4 Others (list)?	☐	☒	
1.2 Is the laboratory currently receiving samples from Libby Superfund Site Operable Units?	☒	☐	

If "**YES,**" complete the following table:

Operable Unit	Matrix/Method(s)	Project/Comments
Various	Soil by PLM-VE (Libby SOP)	
Various	Soil by PLM-GRAV (Libby SOP)	
Various	Air by ISO 10312 & 13794	
Various	Air by AHERA	
Various	Water by EPA 100.1/100.2	
Various	Air by PCM NIOSH 7400	
Various	Bulk by PLM NIOSH 9000	
Various	Dust by ASTM D5755	

2.0 LABORATORY SECURITY	Yes	No	Comments
2.1 Are visitors required to sign in?	☒	☐	
2.2 Are all entrances to the laboratory secured?	☒	☐	

Additional Comments:

3.0 PROJECT INITIATION/PROJECT MANAGEMENT	Yes	No	Comments
3.1 Are there designated project managers or a project management team to ensure samples received are properly processed?	☒	☐	Analytical – Roy Pescador Other – Cathy Lusher
3.2 Are project-specific requirements and procedures communicated to laboratory staff:			
3.2.1 Project-specific SOPs?	☒	☐	All personnel have access to the CDM eRoom.
3.2.2 Laboratory Modifications?	☒	☐	
3.2.3 SAP Analytical Summaries?	☒	☐	
3.2.4 Project-specific Electronic Data Deliverables (EDDs)?	☒	☐	
3.2.5 Other (list)?	☐	☒	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

4.0 SAMPLE RECEIPT, LOG-IN, STORAGE, & TRACKING	Yes	No	Comments
4.1 Is the sample receiving area adequate, clean, and orderly?	☒	☐	

Personnel Interviewed

Name	Title	Experience
Roy Pescador	Laboratory Manager	17 Years

4.2 Sample Receipt			
4.2.1 Is there a sample custodian and designated alternate responsible for sample receipt and log-in?	☒	☐	
4.2.2 Is the custodian or alternate available to receive and log-in samples at any time delivery services are operating?	☒	☐	
4.2.3 Are sample shipping containers opened in a HEPA hood (as necessary) to both minimize personal exposure and safeguard against laboratory contamination?	☒	☐	Hoods are available
4.2.4 Does the sample custodian verify and record the following when inspecting shipments and reviewing documentation:			
4.2.4.1 Presence and condition of custody seals?	☒	☐	Mostly walk-in delivery from various contractors.
4.2.4.2 Presence or absence of Chain-of-Custody (COC) records?	☒	☐	
4.2.4.3 Presence or absence of air bill sticker(s)?	☒	☐	
4.2.4.4 Sample condition?	☒	☐	
4.2.4.5 Presence of packaging or packing material which could compromise samples (i.e., vermiculite & polystyrene)?	☒	☐	
4.2.4.6 Problems/discrepancies between samples, documentation, client requests, etc.?	☒	☐	
4.2.4.7 Bulk and air samples received separately?	☒	☐	
4.2.5 Are COC records signed and dated at the time of sample receipt?	☒	☐	
4.2.6 Is a system in place to ensure laboratory personnel are made aware of project specific requirements?	☒	☐	All personnel have access to the CDM eRoom.
4.2.7 Is a system in place to contact the client in case of absent documentation, or discrepancies between COCs, client requests, etc.?	☒	☐	E-mails
4.2.8 Are subsequent resolutions to problems and discrepancies documented?	☒	☐	
4.3 Sample Identification			
4.3.1 Are sample receipt identification logbooks, or a LIMS, used to log-in samples and assign unique laboratory identification numbers?	☒	☐	LIMS
4.3.1.1 Does the logbook or logging system serve as a direct cross-reference between laboratory ID numbers and client ID numbers?	☒	☐	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

4.0 SAMPLE RECEIPT, LOG-IN, STORAGE, & TRACKING	Yes	No	Comments
4.4 Sample Storage			
4.4.1 Are storage facilities sufficient?	☒	☐	
4.4.2 Is the sample storage area secured to prevent entry of unauthorized personnel?	☒	☐	
4.4.3 Is a logbook or other means used to record sample locations?	☒	☐	
4.4.4 Are samples easy to locate from logbook references?	☐	☒	Refer to Finding No.1 in the Audit Report.
4.5 Sample Tracking			
4.5.1 Is a system in place to keep track of samples entering and leaving the storage, sample preparation, and analysis areas?	☒	☐	
4.5.2 Are the retention and/or disposal of unused portions of samples and prepared samples documented?	☒	☐	
4.5.2.1 Are project-specific retention and/or disposal requirements communicated and followed?	☒	☐	
4.6 Standard Operating Procedures (SOPs)			
4.6.1 Are the applicable laboratory SOPs available and followed by laboratory personnel (list)?	☒	☐	

Document Title	Control No.	Description
QA Manual	Rev. 15	Section 5.8.4

4.7 Document Control:	Yes	No	Comments
4.7.1 Are all logbooks, notebooks, forms, or other laboratory documents legible, accurate, and complete (list)?	☒	☐	

Document Title	Description/Comments
EPA Region 8 Site Investigation Logbook	Used to track the transfer of samples, prepared samples, and both hard copy and electronic deliverables

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

5.0 PHASE CONTRAST MICROSCOPY (PCM)	Yes	No	Comments
5.1 Does the laboratory perform PCM analyses on samples received from the Libby Superfund site? ***If answered "No" precede to Section 6.0 of the checklist.***	NA	NA	
5.2 Is the PCM area adequate, clean, and orderly?	NA	NA	
5.3 Are steps taken to prevent the cross-contamination of equipment, supplies, and reagents?	NA	NA	

Personnel Interviewed

Name	Title	Experience

5.4 Methods and Guidance Documents	Yes	No	Comments
5.4.1 Are the applicable guidance documents available for reference:			
5.4.1.1 NIOSH Method 7400 (Issue 2), 1994?	NA	NA	
5.4.1.2 Other (list)?	NA	NA	
5.4.2 Are project-specific requirements communicated to laboratory personnel and available for reference:			
5.4.2.1 Laboratory Modification LB-000015A?	NA	NA	
5.4.2.2 SOP EPA-Libby-08?	NA	NA	
5.4.2.3 SAP Analytical Summaries?	NA	NA	
5.4.2.4 Project-specific Electronic Data Deliverables (EDDs)?	NA	NA	
5.4.2.5 Other (list)?	NA	NA	
5.5 Equipment			
5.5.1 Ventilation Hoods:			
5.5.1.1 Checked routinely and recorded in a permanent logbook?	NA	NA	
5.5.2 Are the microscopes used to analyze samples equipped with the following:			
5.5.2.1 Positive phase contrast, with green or blue filter?	NA	NA	
5.5.2.2 Adjustable field iris?	NA	NA	
5.5.2.3 Eyepiece (8 to 10X)?	NA	NA	
5.5.2.4 Phase magnification (40 to 45X)?	NA	NA	
5.5.2.5 Walton-Beckett Graticule?	NA	NA	
5.5.2.6 Stage micrometer with 0.01 mm subdivisions?	NA	NA	
5.5.3 Are microscope and phase ring alignment checks conducted daily?	NA	NA	
5.5.4 Is resolution periodically checked using an HSE/NPL slide?	NA	NA	
5.5.5 Are maintenance and calibration activities recorded in microscope-specific logbooks?	NA	NA	

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

5.0 PHASE CONTRAST MICROSCOPY (PCM)	Yes	No	Comments
5.6 Sample Preparation			
5.6.1 Are filters prepared as described in the applicable method(s)?	NA	NA	
5.6.2 Are filters visibly overloaded (>25%) or contain loose debris prepared indirectly as described in SOP EPA-Libby-08?	NA	NA	
5.7 Sample Analysis			
5.7.1 Are the appropriate counting rules used (A or B)?	NA	NA	
5.7.2 How are the fields and fibers tracked and recorded? Calibrated counter is used			
5.8 Quality Control			
5.8.1 Is each analyst provided a minimum of one reference slide per work day?	NA	NA	
5.8.2 Are recounts analyzed at a frequency of 1 per 10 samples analyzed?	NA	NA	
5.8.2.1 For count pairs not within acceptance limits are associated samples recounted?	NA	NA	
5.9 Standard Operating Procedures (SOPs)			
5.9.1 Are the applicable laboratory SOPs available and followed by laboratory personnel (list)?	NA	NA	

Document Title	Control No.	Description

5.10 Document Control	Yes	No	Comments
5.10.1 Are all logbooks, notebooks, forms, or other laboratory documents legible, accurate, and complete (list)?	NA	NA	

Document Title	Description/Comments

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

6.0 TRANSMISSION ELECTRON MICROSCOPY (TEM) GRID PREPARATION	Yes	No	Comments
6.1 Are the grid preparation areas adequate, clean, and orderly?	☒	☐	
6.2 Are bulk samples prepared in an area separate from that used to prepare air and dust samples?	☒	☐	
6.3 Are steps taken to prevent the cross-contamination of equipment, supplies, and reagents?	☐	☒	Refer to Finding No. 2 in the Audit Report

Personnel Interviewed

Name	Title	Experience
Deven Barney	Laboratory Analyst	7 Years

6.4 Equipment & Supplies	Yes	No	Comments
6.4.1 Ventilation Hoods:			
6.4.1.1 Checked routinely and recorded in a permanent logbook?	☒	☐	Checked quarterly.
6.4.2 Drying oven:			
6.4.2.1 Checked routinely and recorded in a permanent logbook?	☒	☐	Oven calibrated to 60ºC.
Note: Desiccator is an option for indirect preparation.			
6.4.3 Muffle furnace:			
6.4.3.1 Checked routinely and recorded in a permanent logbook?	☐	☒	Refer to Finding No. 3 in the Audit Report.
6.4.4 Analytical balances:			
6.4.4.1 Checked routinely and recorded in a permanent logbook?	☐	☒	Refer to Finding Nos. 4 and 5 in the Audit Report.
6.4.4.2 Calibrated within the last 12 months by a certified technician?	☐	☒	
6.4.5 Plasma Asher:			
6.4.5.1 Calibrated at least quarterly and recorded in a permanent logbook?	☒	☐	
Refer to Request for Modification LB-000085A			
6.4.6 Sputter Coater (Vacuum evaporator):			
6.4.6.1 Checked routinely and recorded in a permanent logbook?	☒	☐	
6.4.7 Filtration Apparatus (for indirect preparation):			
6.4.7.1 Are disposable or glass funnels used (record catalogue #)?	☒	☐	Use 25mm disposable funnels from Environmental Express (catalogue #F1500).
6.4.7.2 Has the Effective Filtration Area (EFA) been determined and recorded for each apparatus?	☒	☐	
6.4.8 TEM Grids:			
6.4.8.1 Is documentation for average grid opening determination available?	☒	☐	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

6.0 TRANSMISSION ELECTRON MICROSCOPY (TEM) GRID PREPARATION	Yes	No	Comments
6.5 Direct and Indirect Preparation Methodology			
6.5.1 What method(s) does the laboratory use to prepare air and dust samples for TEM analysis:			
6.5.1.1 40 CFR, Chapter 1, Part 763, Subpart E - AHERA?	☒	☐	
6.5.1.2 ISO 10312:1195 E - Determination of Asbestos Fibers?	☒	☐	
6.5.1.3 ASTM D 5755-09 - Micro vacuum Sampling and Indirect Analysis of Dust by TEM?	☒	☐	
6.5.1.4 Others (list)?	☒	☐	EPA 100.2/100.2
6.5.2 Are project-specific requirements communicated to laboratory personnel and available for reference:			
6.5.2.1 Laboratory Modifications?	☒	☐	All personnel have access to the
6.5.2.2 Project-specific SOPs?	☒	☐	CDM eRoom.
6.5.2.3 SAP Analytical Summaries?	☒	☐	
6.5.2.4 Other (list)?	☐	☒	
6.6 Sample Inspection			
6.6.1 Are air filter cassettes carefully wet-wiped prior to being transferred to the clean preparation area for inspection?	☒	☐	
6.6.2 Are air filter samples which are visibly overloaded, exhibit uneven loading, or contain loose debris, prepared indirectly? *Refer to Laboratory Modifications LB-000016H & LB-000031G*	☒	☐	
6.6.3 Are all ambient air samples dried upon receipt at the on-site laboratory (i.e., EMSL-Libby) prior to preparation and analysis? *Refer to Laboratory Modification LB-000055A*	☒	☐	
6.7 Direct Preparation of MCE and Polycarbonate Filters			
6.7.1 Are MCE filters collapsed using either a Di-Methyl Formamide (DMF) or acetone atmosphere (AA) technique (describe technique)? *The use of an acetone vaporizer ("hot block") is not advised due to the formation of wind rows and tilted fibers.*	☒	☐	DMF/acetone solution.
6.7.2 Is plasma etching performed on collapsed MCE filters?	☒	☐	
6.7.2.1 Is a 5 to10% layer of the collapsed surface removed during etching?	☒	☐	5% is etched.
6.7.3 Are collapsed MCE filters and secured polycarbonate filters transferred to a vacuum evaporator for carbon coating?	☒	☐	
6.7.4 Are excised filter sections placed on the appropriately labeled TEM grids and cleared using a Jaffe Washer or an equivalent technique (describe)?	☒	☐	Cleared with acetone.
6.7.5 Are samples checked for remaining filter residue after clearing?	☒	☐	
6.7.5.1 If residue remains, is condensation washing or an equivalent technique used (describe technique)?	☒	☐	Extend acetone clearing.
Additional Comments:			

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

6.0 TRANSMISSION ELECTRON MICROSCOPY (TEM) GRID PREPARATION	Yes	No	Comments
6.8 Indirect Sample Preparation of Air and Dust Samples			
6.8.1 Are the applicable Libby guidance documents available for reference:			
6.8.1.1 SOP EPA-Libby-08 – Indirect Preparation of Air and Dust Sample for TEM Analysis?	☒	☐	
6.8.2 Sample filtration:			
6.8.3 Are the applicable SAP Analytical Summaries reviewed to determine the whether or not filter samples must be ashed?	☒	☐	
6.8.3.1 Are cassettes examined for loose material?	☒	☐	
6.8.3.1.1 If loose material or uneven loading is not evident, is a portion of the air samples retained?	☒	☐	
6.8.3.1.2 If loose material is evident, is the loose material filtered along with the air filter?	☒	☐	
6.8.3.2 Ashing (if applicable):			
6.8.3.2.1 Are filters covered with aluminum foil and placed in a plasma asher?	☒	☐	
6.8.3.2.2 Is the plasma asher operated at minimum power?	☒	☐	
6.8.3.2.3 Is 100% ashing confirmed by visual observation?	☒	☐	
6.8.3.3 Are air filters, loose material, dust, or ash, rinsed into a beaker and brought to a final volume of 100 mL with particle-free water?	☒	☐	
6.8.3.3.1 Adjusted to a pH of 3-4 with a 10% solution of glacial acetic acid?	☒	☐	
6.8.3.3.2 Sonicated for 3 minutes and allowed to settle for 2 minutes prior to filtering?	☒	☐	
6.8.3.4 Are the appropriate aliquots of filtrate passed through a disposable 25 mm filter assembly with a 0.2 µm MCE filter with a 5.0 µm MCE support pad?	☒	☐	
6.8.4 Are serial dilutions performed as necessary?	☒	☐	
6.8.5 Are TEM grids prepared as described in Section 6.7 of this checklist?	☒	☐	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

6.0 TRANSMISSION ELECTRON MICROSCOPY (TEM) GRID PREPARATION	Yes	No	Comments
6.9 Water Sample Preparation			
6.9.1 What method(s) does the laboratory use to prepare water samples for TEM analysis:			
6.9.1.1 EPA Method 100.2 - Determination of Asbestos Structures Over 10 µm in Length in Drinking Water?	☒	☐	
6.9.1.2 EPA Method 100.1 - Determination of Asbestos Fibers Drinking Water?	☒	☐	
6.9.1.3 Others (describe)? _____	☐	☒	
6.9.2 Are samples received and filtered by the laboratory within 48 hours of collection?	☐	☒	Since samples are treated with UV light and ozone prior to analysis, refrigeration and filtering are not necessary.
6.9.2.1 If not, are they stored in a refrigerator until filtered?	☐	☒	
6.9.3 Laboratory Modification LB-000020A:			
6.9.3.1 Do samples undergo treatment with ozone/UV light?	☒	☐	
6.9.3.2 Are samples hand-agitated and sonicated?	☒	☐	
Refer to Section 6.2 of EPA Method 100.1			
6.9.4 Are the appropriate aliquots of the original sample poured though a 25 mm or 47 mm MCE filter (0.22 µm or smaller pore size) with an MCE filter (5 µm pore size) backing pad? Note: No less than 1 mL must be used as an aliquot.	☒	☐	
6.9.5 Are TEM grids prepared as described in Section 6.7 of this checklist?	☒	☐	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

6.0 TRANSMISSION ELECTRON MICROSCOPY (TEM) GRID PREPARATION	Yes	No	Comments
6.10 OU3 Tree Bark Sample Preparation			
6.10.1 Are the applicable Libby guidance documents available for reference:			
6.10.1.1 EPA-Libby-2012-12 – Sampling and Analysis of Tree Bark for Asbestos?	☒	☐	
6.10.2 Drying and Ashing:			
6.10.2.1 Are the diameter and thickness of the tree bark samples measured and recorded to an accuracy of ± 2mm?	NA	NA	Although the laboratory now has the equipment to prepare tree bark samples for TEM analysis, not all of the equipment (i.e. balance and muffle furnace) have been properly calibrated. This area will be revisited during the 2014 on-site audit.
6.10.2.2 Is the entire tree bark sample weighed and placed in an oven for drying?	NA	NA	
6.10.2.2.1 Dried at 80º C until the weight stabilizes, a minimum of 6 hours, and weighed?	NA	NA	
6.10.2.3 Is the bark sample then covered and placed in a muffle furnace at 450º C for 18 hours, or until all organic matter has been removed, and weighed?	NA	NA	
6.10.2.3.1 Is the furnace ramped from 0º F to 450º C?	NA	NA	
6.10.3 Acid Treatment:			
6.10.3.1 After adding approximately 1-2 mL of DI water, is 10-20 of concentrated HCL added until no further reaction is visible (approx. 3-5 minutes)?	NA	NA	
6.10.3.2 Are samples diluted, transferred to a 100 mL container (with lid) and brought to a final volume of 100 mL with fiber-free DI water?	NA	NA	
6.10.3.3 Are samples capped, inverted 5-6 times, and sonicated for 2 minutes in preparation for filtering?	NA	NA	
6.10.4 Filtration:			
6.10.4.1 Are 5-20 mLs of solution transferred to a second container and brought to a volume of 100 mL with fiber-free DI water?	NA	NA	
6.10.4.2 Are dilutions agitated (inverted 5-6 times) and filtered through a 47 mm MCE filter (0.45 µm pore size)?	NA	NA	
6.10.4.2.1 Are additional dilutions prepared if the loading on the filter appears either too heavy (> 20%) or too light?	NA	NA	
6.10.5 Are TEM grids prepared as described in Section 6.7 of this checklist?	NA	NA	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

6.0 TRANSMISSION ELECTRON MICROSCOPY (TEM) GRID PREPARATION	Yes	No	Comments
6.11 OU3 Duff Sample Preparation			
6.11.1 Are the applicable Libby guidance documents available for reference:			
6.11.1.1 EPA-Libby-2012-11 – Sampling and Analysis of Duff for Asbestos?	☒	☐	
6.11.2 Drying and Ashing:			
6.11.2.1 Are the appropriate number of aluminum trays weighed and tared?	NA	NA	Although the laboratory now has the equipment to prepare duff samples for TEM analysis, not all of the equipment (i.e. balance and muffle furnace) have been properly calibrated. This area will be revisited during the 2014 on-site audit.
6.11.2.1.1 For tracking purposes, is each tray marked with a unique number?	NA	NA	
6.11.2.2 Are trays filled to approximately ¾, dried at 60º C until the weight stabilizes a minimum of 10 hours, and weighed?	NA	NA	
6.11.2.3 Are dried duff samples transferred to covered pans and placed in a muffle furnace at 450º C for 18 hours, or until all organic matter has been removed, and weighed?	NA	NA	
6.11.2.4 Are ashed samples transferred to Zip-lock bags and homogenized?	NA	NA	
6.11.2.4.1 If an individual sample was split between multiple trays, was it combined into one Zip-lock bag?	NA	NA	
6.11.3 Acid Treatment:			
6.11.3.1 After adding approximately 1-2 mL of DI water to 0.25 grams (measured to ± 0.01 g) of ashed sample, is 10-20 mL of concentrated HCL added until no further reaction is visible (approx. 3-5 minutes)?	NA	NA	
6.11.3.2 Are samples diluted, transferred to a 100 mL container (with lid) and brought to a final volume of 100 mL with fiber-free DI water?	NA	NA	
6.11.3.3 Are sample capped, inverted 5-6 times, and sonicated for 2 minutes in preparation for filtering?	NA	NA	
6.11.4 Filtration:			
6.11.4.1 Is 0.1 to 1.0 mL of solution transferred to a second container and brought to a volume of 100 mL with fiber-free DI water?	NA	NA	
6.11.4.2 Are dilutions agitated (inverted 5-6 times) and filtered through a 47 mm MCE filter (0.45 µm pore size)?	NA	NA	
6.11.4.2.1 Are additional dilutions prepared if the loading on the filter appears either too heavy (> 20%) or too light?	NA	NA	
6.11.5 Are TEM grids prepared as described in Section 6.7 of this checklist?	NA	NA	
Additional Comments:			

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

6.0 TRANSMISSION ELECTRON MICROSCOPY (TEM) GRID PREPARATION	Yes	No	Comments
6.12 Grid Preparation/filtrate Storage			
6.12.1 For indirect preparations, are remaining filtrates filtered onto the appropriate filter(s) to be archived?	☒	☐	
6.12.2 Are all remaining filters and filter portions labeled prior to archiving?	☒	☐	
6.12.3 Are grids stored in marked grid storage boxes or other suitable containers and stored in a dust/fiber free environment?	☒	☐	
6.12.4 Is the location of grid preparation recorded in such a manner that they can be retrieved upon request in a timely manner?	☒	☐	
6.13 Quality Control Samples			
6.13.1 Are quality control samples prepared at the described frequency:			
6.13.1.1 Are laboratory blanks (LB) prepared at a frequency of 4% or with each preparation batch, whichever is more frequent?	☒	☐	
6.13.1.2 Are re-preparations prepared at a frequency of 1%?	☒	☐	
6.14 Standard Operating Procedures (SOPs)			
6.14.1 Are the applicable laboratory SOPs available and followed by laboratory personnel (list)?	☒	☐	All SOPs are available on the laboratory E-Link.

Document Title	Control No.	Description

6.15 Document Control	Yes	No	Comments
6.15.1 Are all logbooks, notebooks, forms, or other laboratory documents legible, accurate, and complete (list)?	☒	☐	

Document Title	Description/Comments

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

7.0 TEM ANALYSIS	Yes	No	Comments
7.1 Are TEM areas adequate, clean, and orderly?	☒	☐	
7.2 Are steps taken to prevent the cross-contamination of equipment, supplies, and reagents?	☒	☐	

Personnel Interviewed

Name	Title	Experience
Roy Pescador	TEM Analyst	16 Years

7.3 Methods and Guidance Documents	Yes	No	Comments
7.3.1 What method(s) does the laboratory use to analyze samples TEM:			
7.3.1.1 40 CFR, Chapter 1, Part 763, Subpart E (AHERA)?	☒	☐	
7.3.1.2 ISO 10312:1995 E - Determination of Asbestos Fibers?	☒	☐	
7.3.1.3 ASTM D 5755-09 - Microvacuum Sampling and Indirect Analysis of Dust by TEM?	☒	☐	
7.3.1.4 EPA Method 100.2 - Determination of Asbestos Structures Over 10 µm in Length in Drinking Water?	☒	☐	
7.3.1.5 Others (list)? ASTM 6480	☒	☐	
7.3.2 Are project-specific requirements communicated to laboratory personnel and available for reference:			
7.3.2.1 Laboratory Modifications?	☒	☐	All personnel have access to the CDM eRoom.
7.3.2.2 Project-specific SOPs?	☒	☐	
7.3.2.3 SAP Analytical Summaries?	☒	☐	
7.3.2.4 Project-specific Electronic Data Deliverables (EDDs)?	☒	☐	
7.3.2.5 Other (list)?	☐	☒	
7.4 TEM Instrumentation			
7.4.1 Does TEM instrumentation meet the following requirements:			
7.4.1.1 Capable of being operated at between 80 and 120 kV?	☒	☐	
7.4.1.2 Electron diffraction (ED) and energy dispersive X-ray (EDX) capabilities?	☒	☐	
7.4.1.3 Fluorescent screen with an inscribed or overlaid calibrated scale?	☒	☐	
7.4.2 Are the instruments equipped with thin film or beryllium windows (list below if necessary)? Both (below)	☒	☐	
7.4.3 Are all routine and non-routine maintenance activities recorded in instrument-specific logbooks?	☒	☐	

Instrument No.	Make	Model	Capabilities
27-1	JOEL	JEM-100CX	Thin Film
27-2	JOEL	JEM-100CXII	Beryllium

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

7.0 TEM ANALYSIS	Yes	No	Comments
7.5 Instrument Calibration (Laboratory Modification LB-00085A)			
7.5.1 Is microscope alignment performed daily:			
7.5.1.1 Centering of electron beam?	NA	NA	
7.5.1.2 Electron beam is properly stigmated on either side of crossover?	NA	NA	
7.5.1.3 Image properly focused?	NA	NA	
7.5.2 Is the TEM screen magnification calibrated monthly?	NA	NA	
7.5.3 Is the camera constant calibrated monthly?	NA	NA	
7.5.4 Is the spot size diameter determined to be less than 250 nm quarterly?	NA	NA	
7.5.5 Is the low beam dose (>= 15 seconds for Chrysotile) verified quarterly?	NA	NA	
7.5.6 EDXA System:			
7.5.6.1 Is X-ray energy versus channel for two peaks (i.e., Cu/Al) checked daily?	NA	NA	
7.5.6.2 Is detector resolution (Mn) checked quarterly?	NA	NA	
7.5.6.3 Are K-factors relative to Si determined for Na, Mg, Al, Ca, and Fe quarterly?	NA	NA	
7.5.7 Are instrument calibration records maintained in instrument-specific logbooks?	NA	NA	
7.6 Reference Materials			
7.6.1 Does the laboratory maintain a library of reference materials on asbestos and other fiber types?	NA	NA	
7.6.2 Are instrument-specific “LA” spectra available, posted near the TEM?	☒	☐	Yes, generated in 9/2007.
7.7 Grid Acceptance/Rejection Criteria			
7.7.1 Grid preparation rejection criteria:			
7.7.1.1 The replica is too dark due to poor dissolution?	NA	NA	
7.7.1.2 Replica is doubled or folded?	NA	NA	
7.7.1.3 Replica has > 25% obscuration rejected?	NA	NA	
7.7.1.4 Replica has < 50 intact grid openings?	NA	NA	
Refer to Request for Modifications LB-000016H and LB-000031G			
7.7.2 Are samples associated with grids determined to be overloaded (>25%) re-prepped using the indirect-transfer technique described in SOP EPA-Libby-08?	NA	NA	

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

7.0 TEM ANALYSIS	Yes	No	Comments
7.8 Modifications to AHERA & ASTM D5755:			
7.8.1 Laboratory Modification LB-000031G:			
7.8.1.1 Are structures classified as fibers (F), bundles (B), clusters (C) or matrices (M)?	NA	NA	
7.8.1.2 Are the actual lengths and widths of fibers, bundles, clusters and matrices (M) recorded?	NA	NA	
7.8.1.3 For disperse matrices and clusters, is the length of the longest protruding structure recorded?	NA	NA	
7.8.1.4 Unless identified as a “close call” (LB-000066D), are NAMs not recorded?	NA	NA	
7.8.1.5 Is the designation “ND” used to document when no structures are detected in a grid opening?	NA	NA	
7.8.1.6 Are fibers, bundles, clusters and matrices only recorded they contain individual constituent fibers meeting the aspect ratio criterion?	NA	NA	
7.8.1.7 Are non-countable recorded, but not counted, for informational purposes?	NA	NA	
7.8.1.8 Is the entire length recorded for structures originating in one grid opening and extending to an adjacent grid opening?	NA	NA	
7.8.2 Laboratory Modification LB-000067:			
7.8.2.1 Are the structure identification codes described in Tables D.1 and D.2 of ISO Method 10312 used?	NA	NA	
7.9 Modifications to EPA Method 100.2:			
7.9.1 Laboratory Modification LB-000020:			
7.9.1.1 Are all applicable analyte structures, including those comprising the LA complex, ≥ 0.5 µ in length with a ≥ AR recorded?	NA	NA	
7.9.1.2 Are a maximum of 10 grid openings counted?	NA	NA	
7.9.2 Laboratory Modification LB-000067:			
7.9.2.1 Are the structure identification codes described in Tables D.1 and D.2 of ISO Method 10312 used?	NA	NA	

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

7.0 TEM ANALYSIS	Yes	No	Comments
7.10 Modifications to ISO Method 10312:			
7.10.1 Laboratory Modification LB-000016H:			
7.10.1.1 Unless identified as a “close call” (LB-000066D), are NAMs recorded?	NA	NA	
7.10.1.2 Are bundles only recorded if they contain individual constituent fibers meeting the aspect ratio criterion?	NA	NA	
7.10.1.3 Are bundles, compact clusters, and compact matrices counted regardless of aspect ratio?	NA	NA	
7.10.1.4 Are structures that intersect non-countable grid bars recorded for informational purposes?	NA	NA	
7.10.1.5 Are component structures, which do not intersect non-countable grid bars, but are within non-countable structures counted?	NA	NA	
7.10.1.6 Is the entire length recorded for structures originating in one grid opening and extending to an adjacent grid opening?	NA	NA	
7.10.1.7 For structures which intersect more than one grid bar is the observed length of the structure recorded?	NA	NA	
7.10.1.8 Are the recorded rules for partially obscured structures properly applied (i.e., MFO and MBO)?	NA	NA	
7.10.1.9 Are the counting and recording rules for the identification of PCMe structures at “low magnification” applied?	NA	NA	
7.11 Common TEM Modifications:			
7.11.1 Laboratory Modification LB-000030:			
7.11.1.1 Are highly detailed sketches of up to 50 asbestos structures provided?	NA	NA	
7.11.2 Laboratory Modification LB-000066D:			
7.11.2.1 Is the presence or absence of sodium and potassium recorded for all LA, OA and NAM particles (NaK, NaX, XK or XX)?	NA	NA	
7.11.2.2 Is probable mineral identification code recorded for all particles?	NA	NA	
7.11.2.2.1 Are LA particles identified as WRTA, AC, TR or AT?	NA	NA	
7.11.2.2.2 Are OA particles identified as AM, AN or CR?	NA	NA	
7.11.2.2.3 Are NAMs indicated as PY, OT or UN?	NA	NA	
7.11.2.3 Is one SAED pattern recorded for each amphibole asbestos type encountered per samples?	NA	NA	
7.11.2.4 Are EDS spectrum (a maximum of 5) collected for up to 5 LA and 5 Close-call NAM per sample?	NA	NA	

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

7.0 TEM ANALYSIS	Yes	No	Comments
7.12 Counting/stopping rules:			
7.12.1 Are the Analytical Summaries reviewed to determine the following:			
7.12.1.1 Analytical Sensitivity?	NA	NA	
7.12.1.2 Recording rules (i.e., AR)?	NA	NA	
7.12.1.3 Stopping rules (i.e., abundant CH)?	NA	NA	
7.12.1.4 Applicable Laboratory Modifications?	NA	NA	
7.12.1.5 Investigative or non-investigative?	NA	NA	
7.13 Quality Control Analyses (Laboratory Modification LB-000029C)			
7.13.1 Are quality control samples analyzed at the required frequencies:			
7.13.1.1 Laboratory blanks – Frequency 4%?	NA	NA	
7.13.1.2 Recount Same (RS) - Frequency of 1%?	NA	NA	
7.13.1.3 Recount Different (RD) - Frequency of 2.5%?	NA	NA	
7.13.1.4 Inter-laboratory - Frequency of 0.5%?	NA	NA	
7.13.1.5 Verified Analysis (VA) - Frequency of 1%?	NA	NA	
7.13.1.6 Re-preparations – Frequency of 1%	NA	NA	
7.13.2 Are samples selected for RS, RD and VA analyses in accordance with Laboratory Modification LB-000029C?	NA	NA	
7.13.3 Is the procedure used to evaluate QC sample analyses in accordance with Laboratory Modification LB-000029C?	NA	NA	
7.14 Standard Operating Procedures (SOPs)			
7.14.1 Are the applicable laboratory SOPs available and followed by laboratory personnel (list)?	☒	☐	All SOPs are available on the laboratory E-Link.

Document Title	Control No.	Description

7.15 Document Control	Yes	No	Comments
7.15.1 Are all logbooks, notebooks, forms, or other laboratory documents legible, accurate, and complete (list)?	☒	☐	

Document Title	Description/Comments

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

8.0 POLARIZED LIGHT MICROSCOPY (PLM)	Yes	No	Comments
8.1 Are PLM areas adequate, clean, and orderly?	☒	☐	
8.2 Are steps taken to prevent the cross-contamination of equipment, supplies, and reagents?	☒	☐	
Personnel Interviewed			

Name	Title	Experience
Kelly Colberg	PLM Analyst	6 Years
Margi Carr	PLM Analyst	6 months *

*Margi has been back with EMSL for six month, but has previous PLM experience.

8.3 Methods and Guidance Documents	Yes	No	Comments
8.3.1 Are the applicable guidance documents available for reference:			
8.3.1.1 EPA SOP SRC-Libby-01?	☒	☐	
8.3.1.2 EPA SOP SRC-Libby-03?	☒	☐	
8.3.1.3 NIOSH 9002, Issue 2 - Asbestos (Bulk) by PLM?	☒	☐	
8.3.1.4 Others (list)?	☐	☒	
8.3.2 Are project-specific requirements communicated to laboratory personnel and available for reference:			
8.3.2.1 Laboratory Modifications?	☒	☐	All personnel have access to the CDM eRoom.
8.3.2.2 Project-specific SOPs?	☒	☐	
8.3.2.3 SAP Analytical Summaries?	☒	☐	
8.3.2.4 Project-specific Electronic Data Deliverables (EDDs)?	☒	☐	
8.3.2.5 Other (list)?	☐	☒	
8.4 Equipment			
8.4.1 Ventilation Hoods:			
8.4.1.1 Checked routinely and recorded in a permanent logbook?	☒	☐	
8.4.2 Drying oven (optional):			
8.4.2.1 Checked routinely and recorded in a permanent logbook?	☒	☐	
8.4.3 Muffle furnace:			
8.4.3.1 Checked routinely and recorded in a permanent logbook?	☒	☐	Available, but not in use.

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

8.0 POLARIZED LIGHT MICROSCOPY (PLM)	Yes	No	Comments
8.4.4 Analytical balances:			
8.4.4.1 Two balances:			
8.4.4.1.1 Accurate to 0.01 g, range of 0.01 to 1000 g?	☒	☐	
8.4.4.1.2 Accurate to 1 mg?	☒	☐	
8.4.4.2 Checked routinely and recorded in a permanent logbook?	☒	☐	
8.4.4.3 Calibrated within the last 12 months by a certified technician?	☒	☐	
8.5 Stereomicroscope			
8.5.1 Do stereomicroscopes meet the following requirements:			
8.5.1.1 Magnification range of 10X to 50X?	☒	☐	
8.5.1.2 Incandescent or fluorescent light source?	☒	☐	
8.6 Polarized Light Microscope			
8.6.1 Are PLMs equipped with the following:			
8.6.1.1 Light source and replacement bulbs?	☒	☐	
8.6.1.2 Binocular observation tube?	☒	☐	
8.6.1.3 Blue daylight filter?	☒	☐	
8.6.1.4 Oculars (10X)?	☒	☐	
8.6.1.5 Objectives: 10X, 20X and 40X (or similar)?	☒	☐	
8.6.1.6 10X dispersion staining objective?	☒	☐	
8.6.1.7 A 360 degree graduated rotating stage?	☒	☐	
8.6.1.8 Polarizer and analyzer aligned at 90 degrees to one another?	☒	☐	
8.6.1.9 Bertrand lens?	☒	☐	
8.6.1.10 Substage condenser with iris diaphragm?	☒	☐	
8.6.1.11 Accessory slot for compensator plate?	☒	☐	
8.6.1.12 First order red (550 nanometer) compensator plate?	☒	☐	
8.6.1.13 Crosshair reticle?	☒	☐	
8.6.1.14 Adjustment tools?	☒	☐	
8.6.2 Are microscopes well-maintained, and are all routine and non-routine maintenance activities recorded in instrument-specific logbooks?	☒	☐	

Instrument No.	Make	Model	Capabilities
No. 2	Olympus	BH-2	
No. 1	Leica	Meiji	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

8.0 POLARIZED LIGHT MICROSCOPY (PLM)	Yes	No	Comments
8.7 Refractive Index Liquids			
8.7.1 What refractive index liquids are available:			
8.7.1.1 High dispersion RI liquids from 1.620 to 1.640?	NA	NA	
8.7.1.2 1.550 high dispersion RI liquid?	NA	NA	
8.7.1.3 1.680 to 1.700 RI liquids?	NA	NA	
8.7.2 Are refractive index liquids checked daily for contamination?	NA	NA	
8.7.3 Are refractive index (RI) liquids calibrated monthly using a refractometer or other means (describe)?	NA	NA	
8.8 Reference Materials			
8.8.1 Does the laboratory maintain a library of asbestos and non-asbestos reference materials:			
8.8.1.1 NIST SRM 1866b (Ch, Am and Cr)?	NA	NA	
8.8.1.2 NIST SRM 1867a (Tr, Ac, and An)?	NA	NA	
8.8.1.3 USGS LA PEs:	NA	NA	
8.8.1.3.1 LA 0.2% by mass?	NA	NA	
8.8.1.3.2 LA 1.0% by mass?	NA	NA	
8.8.1.3.3 Other (List)?	NA	NA	
8.8.1.4 Controlled LA asbestos (USGS)?	NA	NA	
8.8.1.5 NIST testing round M12001 (winchite/richterite)?	NA	NA	
8.8.1.6 Non-asbestos (i.e., gypsum, calcite, and fiberglass)?	NA	NA	
8.9 PLM Calibration	**Yes**	**No**	**Comments**
8.9.1 Is PLM alignment performed daily:			
8.9.1.1 Alignment?	NA	NA	
8.9.1.2 Stage and objectives centered?	NA	NA	
8.9.1.3 Optic axis centered?	NA	NA	
8.9.1.4 Alignment of the upper/lower polars?	NA	NA	
8.9.1.5 Centered through substage condenser and iris diaphragm?	NA	NA	
8.9.2 Microscope adjustments verified and recorded prior to sample analyses?	NA	NA	

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

8.0 POLARIZED LIGHT MICROSCOPY (PLM)	Yes	No	Comments
8.10 PLM Analysis by NIOSH Method 9002:			
8.10.1 Does the laboratory perform PLM analyses on samples received from the Libby Superfund site? ***If answered "No" precede to Section 8.11 of the checklist.***	NA	NA	
8.10.2 Are samples visually examined by stereomicroscope for the following:			
8.10.2.1 Color?	NA	NA	
8.10.2.2 Homogeneity?	NA	NA	
8.10.2.3 Texture?	NA	NA	
8.10.3 Which of the following techniques are used to prepare samples for analysis:			
8.10.3.1 Mortar & pestle?	NA	NA	
8.10.3.2 Acid washing?	NA	NA	
8.10.3.3 Ashing?	NA	NA	
8.10.3.4 Solvents?	NA	NA	
8.10.3.5 Other (list)?	NA	NA	
8.10.4 For non-friable, organically bound samples requiring ashing and/or acid reduction, are all necessary weights and tare weights measured and recorded?	NA	NA	
8.10.5 Are slides prepared using the appropriate refractive index liquid(s) and scanned for asbestos fibers using the following optical properties:			
8.10.5.1 Morphology?	NA	NA	
8.10.5.2 Color?	NA	NA	
8.10.5.3 Refractive indices?	NA	NA	
8.10.5.4 Pleochroism?	NA	NA	
8.10.5.5 Birefringence?	NA	NA	
8.10.5.6 Extinction characteristics?	NA	NA	
8.10.5.7 Sign of elongation?	NA	NA	
8.10.5.8 Dispersion staining characteristics?	NA	NA	
8.10.6 Are the observed optical properties compared to Table 1 (Optical Properties of Asbestos Fibers) to determine the asbestos mineral present?	NA	NA	
8.10.7 Is a quantitative assessment of asbestos content made from both the gross and microscopic examinations?	NA	NA	
8.10.8 If no fibers are detected in a homogeneous samples are at least two additional slides prepared and analyzed prior to concluding no asbestos is present?	NA	NA	
8.10.9 Is at least one optical property recorded for fibers determined to be non-asbestos fibers?	NA	NA	

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

8.0 POLARIZED LIGHT MICROSCOPY (PLM)	Yes	No	Comments
8.11 PLM-VE (SOP SRC-Libby-03)			
8.11.1 Stereomicroscopic Examination:			
8.11.1.1 Are all sample preparation activities performed within a HEPA-filtered hood?	NA	NA	
8.11.1.2 Is the entire sample transferred to an asbestos-free substrate for examination?	NA	NA	
8.11.1.3 Is the entire sample examined for homogeneity and the presence of suspect fibers?	NA	NA	
8.11.1.4 Are suspect fibers removed with fine forceps and mounted in the appropriate RI liquid for PLM analysis?	NA	NA	
8.11.1.5 Are the stereomicroscopic findings recorded:			
8.11.1.5.1 Sample appearance?	NA	NA	
8.11.1.5.2 Estimated percentage of LA?	NA	NA	
8.11.1.5.3 Estimated percentage of other asbestos types?	NA	NA	
8.11.2 Determination of Ashing the Sample:			
8.11.2.1 Are soil sample containing a significant amount of artifacts ashed prior to being prepared for random PLM mounts?	NA	NA	
8.11.2.1.1 Are samples ashed in a muffle furnace at approximately 480°C?	NA	NA	
8.11.2.1.2 Are the necessary gravimetric measurements recorded for the determination of “Pre-ash percent asbestos”?	NA	NA	
8.11.3 Slide Preparation for PLM-VE:			
8.11.3.1 Are a minimum of five random sub-samples mounted in the appropriate RI liquid (1.620-1.640) for measurement of LA optical properties?	NA	NA	
8.11.4 Supplemental Stereomicroscopic Evaluation:			
8.11.4.1 Following the random slide mount preparation, is the container agitated to cause the particulate to settle and asbestos fibers sort to the surface?	NA	NA	
8.11.4.2 Is the sample re-examined and the fiber pick procedure repeated?	NA	NA	

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

8.0 POLARIZED LIGHT MICROSCOPY (PLM)	Yes	No	Comments
8.11.5 Classification of Asbestos Mineral Type:			
8.11.5.1 Using PLM is entire area of each prepared slide examined for asbestos, non-asbestos and matrix material?	NA	NA	
8.11.5.2 Is positive identification determined from the following six optical properties:			
8.11.5.2.1 Habit?	NA	NA	
8.11.5.2.2 Color & pleochroism (if present)?	NA	NA	
8.11.5.2.3 Both alpha and gamma Refractive indices?	NA	NA	
8.11.5.2.4 Birefringence?	NA	NA	
8.11.5.2.5 Extinction angle?	NA	NA	
8.11.5.2.6 Sign of elongation (positive-slow or negative fast)?	NA	NA	
8.11.5.3 Based on the optical properties, is asbestos classified into one of three categories:			
8.11.5.3.1 Libby Amphibole (LA)?	NA	NA	
8.11.5.3.2 Other Amphibole (OA)?	NA	NA	
8.11.5.3.3 Chrysotile (CH)?	NA	NA	
8.11.5.4 Is at least one optical property recorded for observed non-asbestos fibers?	NA	NA	
8.11.6 Quantification of Asbestos Content:			
8.11.6.1 Is asbestos reported as either mass or area percent for LA?	NA	NA	
8.11.6.2 Are other, non-LA, asbestos types reported in area percent?	NA	NA	
8.11.6.3 Are reference materials used to aid in visual estimation:			
8.11.6.3.1 LA PE reference materials (0.2% or 1.0%)?	☒	☐	
8.11.6.3.2 Are visual estimates of greater than 1% LA performed using calibration standards made in-house from NIST SRMs and NIST PEs?	☒	☐	
8.11.7 Are calibrated visual estimates determined from both the detailed stereomicroscopic observations and examination of the total area for all five random slide mounts?	NA	NA	
8.11.8 Are LA results reported in the appropriate bin categories:			
8.11.8.1 Non-detects recorded as Bin A?	NA	NA	
8.11.8.2 Less than 0.2% LA recorded as Bin B1?	NA	NA	
8.11.8.3 Greater than 0.2%, but less than 1% recorded as Bin B2?	NA	NA	
8.11.8.4 Equal to or greater than 1% recorded as Bin C, with the percentage recorded as a whole number?	NA	NA	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

8.0 POLARIZED LIGHT MICROSCOPY (PLM)	Yes	No	Comments
8.12 PLM-GRAV (SOP SRC-Libby-01)			
8.12.1 Stereomicroscopic Examination:			
8.12.2 Is the entire sample weighed and placed in an appropriate container?	NA	NA	
8.12.3 Does the stereomicroscopic examination include:			
8.12.3.1 Examination of multiple fields of view over the entire sample?	NA	NA	
8.12.3.2 Probing of the sample and breaking clumps where possible?	NA	NA	
8.12.3.3 Manipulation of the sample with the appropriate tools?	NA	NA	
8.12.3.4 Observation homogeneity, texture, friability, color and extent of any asbestos content?	NA	NA	
8.12.4 Doe the analyst refrain from segregating and weighing particles smaller than 2 - 3 mm (1/10 inch)?	NA	NA	
8.12.5 If no particles larger than 2 – 3 mm or larger are present, are one of the following recorded:			
8.12.5.1 No asbestos detected (ND)?	NA	NA	
8.12.5.2 Trace levels of asbestos observed, but not quantified (Tr)?	NA	NA	
8.12.6 Examination by PLM:			
8.12.7 Are tentatively identified asbestos particles examined by PLM as described in SOP SRC-Libby-03 (Section 8.12 of this checklist)?	NA	NA	
8.12.8 If asbestos particles are determined to be OA, are they further characterized:			
8.12.8.1 Amosite (AMOS)?	NA	NA	
8.12.8.2 Anthophylite (ANTH)?	NA	NA	
8.12.8.3 Crocidolite (CROC)?	NA	NA	
8.12.8.4 Unknown (UNK)?	NA	NA	
8.12.9 Is the total weight of each type of positively identified asbestos measured and recorded?	NA	NA	
8.12.10 Record Keeping:			
8.12.11 Is the data log sheet provided in Attachment 1 of the SOP used to record weights the initial (coarse fraction) and segregated asbestos?	NA	NA	

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

8.0 POLARIZED LIGHT MICROSCOPY (PLM)	Yes	No	Comments
8.13 Quality Control Analyses			
8.13.1 Are the following types of QC analyses performed at the required frequencies:			
8.13.1.1 Laboratory duplicate self-check (LDS) at a frequency of 2%?	☒	☐	
8.13.1.2 Laboratory duplicate cross-check (LDC) at a frequency of 8%?	☒	☐	
8.13.2 For sample containing LA, are LDS and LDC analyses considered acceptable if:			
8.13.2.1 For LA results, within 1 Bin category?	NA	NA	
8.13.2.2 For LA results, %LA ≤1%?	NA	NA	
Note: For LA results greater than 1%, the laboratory should refer to their internal QA/QC system.			
8.13.3 Is the appropriate correction action taken when LDC or LDS analyses do not meet acceptance criteria (describe)?	NA	NA	
8.14 Standard Operating Procedures (SOPs)			
8.14.1 Are the applicable laboratory SOPs available and followed by laboratory personnel (list)?	☒	☐	All SOPs are available on the laboratory E-Link.

Document Title	Control No.	Description

8.15 Document Control	Yes	No	Comments
8.15.1 Are all logbooks, notebooks, forms, or other laboratory documents legible, accurate, and complete (list)?	NA	NA	

Document Title	Description/Comments

Additional Comments:

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

9.0 DATA MANAGEMENT	PCM	TEM	PLM	Comments
9.1 Data Package Review and Assembly	**Yes**	**Yes**	**Yes**	
9.1.1 Are deliverables reviewed to ensure project-specific requirements are adhered to:				
9.1.1.1 Request for Modifications to Laboratory Activities?	NA	NA	NA	
9.1.1.2 Project-specific SOPs?	NA	NA	NA	
9.1.1.3 SAP Analytical Summaries?	NA	NA	NA	
9.1.1.4 Project-specific Electronic Data Deliverables (EDDs)?	NA	NA	NA	
9.1.1.5 Other (list)? _____	NA	NA	NA	
9.1.2 Are all deliverables reviewed for completeness and accuracy prior to being submitted:				
9.1.2.1 Hard copy deliverables?	NA	NA	NA	
9.1.2.2 Electronic deliverables?	NA	NA	NA	
9.1.3 Are all reviews documented?	NA	NA	NA	
9.2 Data Submission				
9.2.1 Is the submittal of electronic deliverables tracked and recorded:				
9.2.1.1 Date submitted?	NA	NA	NA	
9.2.1.2 Recipient?	NA	NA	NA	
9.2.2 Is the submittal of hard copy deliverables tracked and recorded:				
9.2.2.1 Date submitted?	NA	NA	NA	
9.2.2.2 Recipient?	NA	NA	NA	
9.3 Data Storage and Archiving				
9.2.3 Are electronic files archived onto suitable media on a frequent basis? How often? _____	NA	NA	NA	
9.2.4 Are all hardcopy data stored in a secured location with limited access (e.g., locking file cabinet)?	NA	NA	NA	

Additional Comments: Since this was a follow-up audit and there were no deficiencies identified in this area from the previous audit, this laboratory area was not evaluated.

LIBBY-SPECIFIC ASBESTOS LABORATORY ON-SITE AUDIT CHECKLIST

USEPA Date(s) of On-site:7/9/2013

10.0 QUALITY ASSURANCE/QUALITY CONTROL	PCM	TEM	PLM	Comments
10.1 Laboratory Certifications	**Yes**	**Yes**	**Yes**	
10.1.1 Is the laboratory accredited for asbestos analysis under the National Voluntary Laboratory Accreditation Program (NVLAP):				
10.1.1.1 Asbestos Fiber Analysis (TEM Method)?	NA	☒	NA	Expires 9/13
10.1.1.2 Asbestos Fiber Analysis (PLM Method)?	NA	NA	☒	Expires 9/13
10.1.2 Is the laboratory accredited for asbestos analysis under the American Industrial Hygiene Association (AIHA), and does it participate in the National Institute for Occupational Safety and Health (NIOSH) Proficiency Analytical Testing (PAT) Program?	NA	NA	NA	
10.2 Training				
10.2.1 Have all analysts undergone training on the proper usage of the equipment and instrumentation used in the respective areas?	NA	NA	NA	
10.2.2 Have all analysts demonstrated proficiency through the preparation and/or analysis of standards or samples of known values?	NA	NA	NA	
10.2.3 Are training records maintained in analyst-specific files?	NA	NA	NA	
10.3 Internal Audits				
10.3.1 Are internal audits conducted on an annual basis using an appropriate checklist?	☒	☒	☒	
10.3.1.1 Are internal audit reports available for review?	☒	☒	☒	
10.4 Corrective/Preventive Action:				
10.4.1 Can the laboratory demonstrate the sequence of problem identification, corrective action, and resumption of duties?	☒	☒	☒	
10.5 Quality Records				
10.5.1 Are SOPs available in the applicable areas for all laboratory-specific procedures?	☒	☒	☒	All SOPs are available on the laboratory E-Link.
10.5.2 Does the laboratory have a Quality Assurance Manual/Plan?	☒	☒	☒	
10.5.3 Does the laboratory compile monthly quality assurance/quality control reports?	☒	☒	☒	
10.6 Environmental Controls/Laboratory Monitoring				
10.6.1 Does the laboratory conduct an environmental monitoring program?	☒	☒	☒	
10.6.2 Is quarterly air monitoring performed in all laboratory areas?	☒	☒	☒	
10.6.2.1 Are the collected samples analyzed by TEM with a target analytical sensitivity of 0.005 structures/cc?	☒	☒	☒	
10.6.2.2 If LA is detected, are the affected areas thoroughly cleaned and a new set of samples collected and analyzed?	☒	☒	☒	
Laboratory Modification LB-000085A				
Additional Comments:				

ATTACHMENT 2

EMSL Analytical (Libby, Montana) Issues and Concerns

CB&I
2700 Chandler Avenue, Building C
Las Vegas, NV 89120
Tel: +1 702 795 0515
Fax: +1 702 795 8210
www.CBI.com

Memorandum

From: Michael P. Lenkauskas
Date: September 20, 2013
Subject: EMSL Analytical (Libby, MT) Issues and Concerns

The following is a summary of issues and analytical data discrepancies associated with the EMSL Analytical laboratory in Libby, Montana. The discrepancies, identified by CDM Smith and CB&I, raise concerns about the quality of the data provided by this laboratory for samples collected from Operable Unit 3 (OU3) of the Libby Superfund Site. The specific issues include:

- TEM Inter-lab sample preparation issues
- Inadequate frequency of project-specific QC analyses
- Possible misidentification of samples
- Result discrepancies between TEM rapid TAT and full analysis of OU3 water samples

TEM Inter-lab Sample Preparation Issues

The EMSL Analytical laboratory experienced an unusually high percentage (37%) of damaged grid openings (GOs) on re-preparations prepared for the 2010 and 2011 TEM inter-laboratory, which resulted in these samples having to be re-prepped again, slowing down what turned out to be an already laborious process. Upon identification of this issue on March 25, 2013, the laboratory was directed to investigate and apply the necessary correction actions prior to preparing the re-preparations for the 2012 TEM inter-laboratory study about to be initiated. The root cause of the damaged GOs, as determined by the laboratory, is described in the attached corrective action (CAR# 1303-1), was the following:

- Grid opening size, EMSL uses a custom made grid with an opening of 0.0130 sq. mm;
- Grid condition;
- Carbon coating thickness;
- Ash time; and
- Packaging and shipping.

In addition to using a grid with a smaller grid opening size (15x15 grids with a G.O.A. of 0.0064 sq. mm) the laboratory is now also pre-cleaning the grids and has adjusted the asher and carbon coating settings. Since none of the 11 samples re-prepped by the laboratory for the 2012 TEM inter-laboratory were received damaged, the corrective actions initiated by the laboratory appear to have resolved the issue.

It should also be noted that undissolved filter material has also been observed on EMSL grid preparations, which will be investigated on a laboratory-by-laboratory basis during the 2012 laboratory on-site audits.

Inadequate Frequency of Project-specific QC Analyses

A review of the QC analyses available in the OU3 database for samples analyzed in 2012 revealed that the frequency at which QC analyses were performed for both TEM and PLM analyses during this period was not in accordance with the criteria described in Laboratory Modification LB-000029D and SOP SRC-

Libby-03 (rev. 3) for TEM and PLM, respectively. The following table provides a summary of analyses performed, the required frequency, the number of QC analyses that should have been performed, and the actually number and percentage of QC analyses that were performed:

Method	QC Type	Sample Analyses	Required Frequency	Performed	Actual Frequency
TEM	LB	293	4%	7	2.4%
TEM	RS	293	1%	0	0%
TEM	RD	293	2.5%	3	1%
TEM	VA	293	1%	2	0.7%
TEM	RP	293	1%	5	1.7%
PLM	LDC	65	8%	1	1.5%
PLM	LDS	65	2%	3	4.6%

Although QC analyses were not performed at the required frequency on a project-specific basis (OU3), they were prepared at the required frequency for all of the operable units combined. This discrepancy was brought to the attention of EMSL Analytical Management on May 22, 2013, who performed an investigation and determined that separate QC logbooks were maintained up until June 25, 2012, at which time they were combined[1]. Effective May 23, 2013 samples received from OU3 are once again recorded in a separate, OU3-specific, QC logbook, ensuring that project-specific QC will be performed at the required frequencies.

Possible Misidentification of Samples

A review of the results from surface water samples collected from OU3 during the spring of 2012 and analyzed by the laboratory indicates that samples were misidentified either in the field during collection or in the laboratory while being processed. Samples possibly misidentified are summarized in the following table:

Index ID	Sample Type	Date Prepared	Date Analyzed	Structures	Comments
P5-10013	Field Sample	5/09/12	5/25/12	0	Same preparation batch.
P5-10014	Field Blank		5/26/12	25	
P5-10067	Field Sample	6/20/12	6/26/12	25	Field sample/field duplicate pair
P5-10068	Field Duplicate		6/26/12	1	
P5-20018	Field Sample	5/17/12	6/01/12	0	Field sample/field duplicate pair. Lab RP had 50 structures.
P5-20019	Field Duplicate		6/02/12	65	
P5-20085	Field Sample	7/04/12	7/09/12	5	Field sample/field duplicate pair
P5-20087	Field Duplicate		7/09/12	27	
P5-20225	Field Sample	9/20/12	11/08/12	25	Field sample/field duplicate pair
P5-20226	Field Duplicate		11/08/12	62	

Although sometimes analyzed on separate days, each of the sample pairs in question were prepared on the same days by the same preparer, increasing the possibility that the misidentification of at least the field duplicate pairs at the laboratory. It should also be noted that with the exception of the sample pair prepared and analyzed in September and November, respectively, which has results that may or may not indicate the samples were misidentified, the remaining samples, which exhibit much greater disparity, were all prepared and analyzed during the spring/early summer 2012.

The potential that the misidentification of samples was brought to EMSL Analytical Management's attention, and on February 19, 2013 the laboratory provided a memo to both EPA and Remedium summarizing the findings of their investigation. The first section of this memo discusses the TEM Rapid TAT versus TEM full analysis discrepancies, which are discussed below. Concerning the possible misidentification of samples, the laboratory offered the explanation that at the time of the misidentifications the laboratory was operating beyond its capacity, creating a disorganized environment

[1] Note that this timeframe coincides with the change in the OU3 laboratory subcontracting mechanism from Remedium to TechLaw.

with staff trying to handle too many responsibilities. Procedural changes put in place by the laboratory to prevent similar situations for occurring in the future include:

- Expansion of the sample preparation area creating a less cluttered workspace in which to stage more samples in an organized manner
- Restricting the number of jobs being prepared simultaneously
- Having one individual track the progress of each individual lab job
- Provide training and improve intra-laboratory communication to better handle lab capacity issues

Note: Although this memorandum indicated that the capabilities of the Denver laboratory were to be increased to handle duff and water samples, as of the spring of 2013, this action has not been implemented.

Result discrepancies between TEM rapid TAT and full analysis of OU3 water samples

For a subset of the Kootenai River water samples collected in 2012, the EMSL-Libby laboratory was requested to perform a "rapid" TAT analysis. This analysis was performed using the same preparation techniques and counting rules as the traditional "full" analysis, but only required the analyst to record the total number of countable LA structures per GO (i.e., recording of structure-specific attributes, such as length, width, and structure type, was not required) to facilitate the faster reporting of water concentrations. Following the rapid TAT analysis, each water sample was subsequently re-analyzed[2] using the traditional full analysis reporting requirements.

A comparison of the rapid vs. full analysis results performed in January/February 2013 revealed significant discrepancies between the reported water concentrations for several samples (examples provided below):

Index ID	Total LA Water Conc. (MFL)	
	Rapid Analysis	Full Analysis
P5-10004	3.7	0
P5-10010	97	0
P5-10008	62	0
P5-10013	40	0

These discrepant results were brought to EMSL Analytical Management's attention, and the laboratory repeated the rapid and full analysis for a subset of the Kootenai River water samples (from the raw water that was in archive) to identify the nature of these discrepancies. The results of these repeated analyses indicated that the reported water concentrations from the original rapid analysis were not confirmed, but that the original full analysis results were confirmed *for most samples*. On this basis, the laboratory provided a memo to both EPA and Remedium on February 19, 2013, recommending that "*all rapid results should be disregarded in favor of the full ISO analyses*". This memo did not specify the reason for the differences between the rapid and full analysis results, but EMSL noted in a subsequent memo on September 4, 2013, that the analyst performing the rapid analysis erroneously utilized PCM recording rules, resulting in the recording of diatom fragments as countable structures.

However, as noted above, the repeat full analyses did not confirm the results for all samples. In particular, for a subset of samples, the repeat full analysis did not confirm either the original rapid analysis or the full analysis:

Index ID	Total LA Water Conc. (MFL)		
	Original Analysis		Repeat
	Rapid Analysis	Full Analysis	Full Analysis
P5-10018	78	35	0
P5-10017	37	58	0

[2] Because the grids from the rapid analysis were often blown due to the original examination, this re-analysis was performed using a newly prepared set of grids from the original filter.

Index ID	Total LA Water Conc. (MFL)		
	Original Analysis		Repeat
	Rapid Analysis	Full Analysis	Full Analysis
P5-10015	34	60	0

In the case of one field blank (P5-10014), the re-analysis supported the unexpected results of the original full analysis, which reported a total LA water concentration of about 25 MFL. Because of these discrepancies, the validity of the original full analysis results is also uncertain.

Resolution of discrepancies for OU3 water samples

Re-analyses of samples collected in 2012

As a consequence of the discrepancies discussed above, several re-analyses were performed of the water samples collected in 2012 from the Phase V Part A (Kootenai) and Part B (Ecological) studies to confirm the originally reported results. This re-analysis effort included the analysis of a subset of water samples from the Kootenai River study (i.e., samples collected during Rounds 1 through 5 from stations LRC-6 and UKR-0) and the in-stream fish toxicity tests (i.e., a subset of the LRC surface water samples from the eyed egg study and 20% of the surface water samples from the fry study). These re-analyses were performed by EMSL-Cinnaminson in July/August 2013 from the raw water[3].

Table 1 (see below) summarizes these results. As shown, of the 25 samples that were re-analyzed, there were 9 samples where the repreparation analysis performed by EMSL-Cinnaminson was statistically different from the original analysis performed by EMSL-Libby (based on a Poisson ratio comparison test at a 90% confidence interval). This means that the difference in LA water concentrations between the original analysis and the repreparation analysis was more than can be attributed to Poisson counting error alone. For the 4 samples that were different from the Part A program (Kootenai), these results confirmed that some type of filter mix-up had occurred for samples P5-10014, P5-10015, P5-10017, and P5-10018 during the original analysis at EMSL-Libby. For sample P5-10014, the results confirmed that both the original analysis (reported in May 2012) and the re-analysis (performed in May 2013) by EMSL-Libby were in error. All of these samples were prepared by the same person on the same day (5/9/2012). This preparation batch included 16 samples (P5-10013 through P5-10027). Most of the samples in this preparation batch (P5-10019 through P5-10027) were associated with a pilot study to evaluate differences in three different water sampling methodologies and were not part of the Kootenai River sampling program.

For the other 5 samples that were different from the Part B program (Ecological), there appears to be a consistent bias, with EMSL-Cinnaminson reporting higher concentrations than EMSL-Libby. Although for most of these samples, the concentrations are usually within a factor of about 3, there was one sample (P5-20027) where the reported concentration by EMSL-Cinnaminson is about 90 times higher than what was reported by EMSL-Libby, which may indicate another potential filter mix-up.

Re-analyses of samples collected in 2013

In addition, approximately 20% of the water samples collected as part of the 2013 eyed egg study were also be randomly selected *a priori* for re-analysis by EMSL-Cinnaminson in July/August 2013. These re-analyses were performed from either the originally prepared filter or the raw water (depending upon the nature of the archived sample).

Table 2 (see below) summarizes these results. A total of 17 samples were selected for re-analysis by EMSL-Cinnaminson; 10 samples were reprepared from the filter (filter was prepared by EMSL-Libby) and 7 samples were reprepared from the raw water. As shown, 8 of the 17 samples that were re-analyzed by EMSL-Cinnaminson was statistically different from the original analysis performed by EMSL-Libby (based on a Poisson ratio comparison test at a 90% confidence interval). Similar to what was observed in

[3] For two samples, the re-analysis was performed from the original filter because no raw water remained (these samples are indicated in the table).

the 2012 re-analyses, there appears to be a consistent bias, with concentrations reported by EMSL-Libby tending to be lower than those reported by EMSL-Cinnaminson. However, concentrations in most samples were usually within a factor of about 2.

Of particular interest are the results for samples P5-20325 and P5-20326. These two samples were preferentially selected for re-analysis because the originally reported LA concentrations suggested that the results for the pore water and its paired surface water got mixed up. The re-analysis performed by EMSL-Cinnaminson confirmed that a filter mix up did occur and that it happened in EMSL-Libby when reporting the results (not in the field)[4].

Conclusions

The results of these re-analyses support the conclusion that filter mix-ups occurred at EMSL-Libby both in 2012 and 2013. The largest mix-up appears to be associated with the set of filters that were prepared during Round 3 of the Phase V, Part A (Kootenai) sampling effort (which included P5-10014, P5-10015, P5-10017, and P5-10018). However, other filter mix-ups outside of this timeframe were also noted, and even occurred during the 2013 study after corrective actions were to have been implemented.

The re-analyses also show that there are differences between the EMSL laboratories in the identification and recording of LA structures in water samples from OU3, albeit the magnitude of the differences in the reported water concentrations are not large (usually within a factor of 2-3).

Resolutions and Recommendations

Based on discussions with EPA, the following resolutions were reached with regard to the 2012/2013 water analyses:

- For samples where the re-analysis confirmed that a filter mix-up occurred (i.e., P5-10014, P5-10015, P5-10017, P5-10018, P5-20325, and P5-20326), the original EMSL-Libby results will be rejected; a corrected EDD will be submitted changing the *Filter Status* field from 'Analyzed' to 'Cancelled' and an analysis comment will be added regarding the rejected status. A modified EDD will be submitted for the corresponding EMSL-Cinnaminson analyses that will be used in preference; a corrected EDD will be submitted changing the *Lab QC Type* from 'Repreparation' to 'Not QC' and an analysis comment will be added explaining why the QC status was changed. The revised EDDs will be uploaded to the OU3 project database.

- For all other samples that were re-analyzed, the EMSL-Libby result will be retained as the 'Not QC' analysis and the EMSL-Cinnaminson result will be retained as the 'Repreparation'. When these results are summarized, the results of the repreparations will be used to demonstrate the between-laboratory differences in TEM counting and recording and results uncertainty/variability, but will not be used to alter the reported results.

For future OU3 investigations, the following recommendations were made:

- Ensure that SAP/QAPPs for 2014 water sampling at OU3 include a 20% repreparation requirement (from raw water) by EMSL-Cinnaminson.

- Ensure that a copy of the analytical summary sheet is included with all submitted chain of custody forms.

- Ensure that all analysts have access to the appropriate eRooms and are familiar with any site-specific methods and procedures prior to analysis.

[4] As shown in the table, EMSL-Cinnaminson performed an extra repreparation analysis which confirmed their results for sample P5-20326.

Additionally, EPA’s laboratory support contractor, Tech Law, Inc., was tasked with providing onsite re-training of all TEM analysts in the EMSL-Libby laboratory, developing a training procedure for all TEM laboratories, and preparing reference material standards (e.g., pyroxene, actinolite, tremolite) to minimize potential between-laboratory differences in LA structure reporting in future TEM analyses.

TABLE 1
LIBBY OU3: 2012 PHASE V, SURFACE WATER RE-ANALYSIS RESULTS
REPREPARATION RESULT COMPARISON

Investigation	Repreparation Type	Index ID	Original Analysis (2012)				Repreparation Analysis (Jul/Aug 2013, EMSL-Cinnaminson)				Poisson Rate Comparison (90% CI)
			Laboratory	Total LA Structures	Sensitivity (1/L)	Total LA Conc (MFL)	Laboratory	Total LA Structures	Sensitivity (1/L)	Total LA Conc (MFL)	
2012 Phase V Part A Surface Water	Raw water	P5-10005	EMSL27	2	3.3E+05	**0.7**	EMSL04	0	1.3E+06	**0**	[0-13.62] The rates are not different
	Raw water	P5-10006	EMSL27	121	3.5E+06	**419**	EMSL04	39	1.2E+07	**473**	[0.65-1.23] The rates are not different
	Raw water	P5-10011	EMSL27	0	1.6E+05	**0**	EMSL04	0	6.4E+05	**0**	Both counts are 0; the rates are not different
	Raw water	P5-10012	EMSL27	27	1.5E+06	**42**	EMSL04	25	1.9E+06	**47**	[0.54-1.46] The rates are not different
	Raw water	**P5-10014**	**EMSL27**	**25**	**9.2E+05**	**23**	**EMSL04**	**0**	**2.6E+04**	**0**	**[0-0] Rate 1 is greater than Rate 2**
	Filter	**P5-10015**	**EMSL27**	**26**	**2.3E+06**	**60**	**EMSL04**	**0**	**1.8E+05**	**0**	**[0-0.01] Rate 1 is greater than Rate 2**
	Raw water	**P5-10017**	**EMSL27**	**25**	**2.3E+06**	**58**	**EMSL04**	**1**	**6.4E+05**	**0.6**	**[17.65-1828.91] Rate 1 is greater than Rate 2**
	Raw water	**P5-10018**	**EMSL27**	**25**	**1.4E+06**	**35**	**EMSL04**	**0**	**6.4E+05**	**0**	**[0-0.06] Rate 1 is greater than Rate 2**
	Raw water	P5-10025	EMSL27	27	2.8E+06	**75**	EMSL04	26	1.9E+06	**51**	[0.91-2.42] The rates are not different
	Raw water	P5-10033	EMSL22	1	4.9E+04	**0.05**	EMSL04	3	2.8E+04	**0.09**	[0.02-5.24] The rates are not different
	Raw water	P5-10034	EMSL22	121	2.8E+05	**33**	EMSL04	114	2.7E+05	**31**	[0.87-1.36] The rates are not different
	Raw water	P5-10053	EMSL04	0	5.0E+04	**0**	EMSL04	1	2.1E+04	**0.02**	[0-44.2] The rates are not different
	Raw water	P5-10056	EMSL04	66	2.5E+05	**16**	EMSL04	84	2.4E+05	**20**	[0.6-1.05] The rates are not different
2012 Phase V Part B Eyed Egg Surface Water	Raw water	P5-20002	EMSL27	58	6.9E+05	**40**	EMSL04	26	1.6E+06	**42**	[0.63-1.46] The rates are not different
	Raw water	P5-20006	EMSL27	33	6.9E+05	**23**	EMSL04	26	8.1E+05	**21**	[0.68-1.74] The rates are not different
	Raw water	P5-20011	EMSL27	25	7.9E+04	**2**	EMSL04	25	2.6E+05	**7**	[0.18-0.5] Rate 1 is less than Rate 2
2012 Phase V Part B Fry Surface Water	Raw water	P5-20016	EMSL04	46	8.2E+05	**38**	EMSL04	60	8.2E+05	**49**	[0.54-1.08] The rates are not different
	Filter	P5-20018	EMSL04	0	8.5E+04	**0**	EMSL04	0	8.6E+04	**0**	Both counts are 0; the rates are not different
	Raw water	P5-20021	EMSL04	26	5.0E+05	**13**	EMSL04	25	5.5E+05	**14**	[0.58-1.58] The rates are not different
	Raw water	P5-20027	EMSL04	25	6.7E+04	**2**	EMSL04	60	2.4E+06	**146**	[0.01-0.02] Rate 1 is less than Rate 2
	Raw water	P5-20031	EMSL04	41	2.5E+05	**10**	EMSL04	73	2.4E+05	**18**	[0.4-0.79] Rate 1 is less than Rate 2
	Raw water	P5-20042	EMSL22	34	1.0E+06	**34**	EMSL04	39	9.7E+05	**38**	[0.59-1.35] The rates are not different
	Raw water	P5-20045	EMSL27	2	5.2E+04	**0.1**	EMSL04	3	5.1E+04	**0.2**	[0.08-4.34] The rates are not different
	Raw water	P5-20069	EMSL27	79	2.8E+05	**22**	EMSL04	42	9.7E+05	**41**	[0.39-0.75] Rate 1 is less than Rate 2
	Raw water	P5-20081	EMSL27	25	1.4E+05	**3**	EMSL04	31	7.8E+05	**24**	[0.09-0.23] Rate 1 is less than Rate 2

All filters pass the CHISQ test for filter loading evenness.

Original Analysis > Repreparation Analysis
Original Analysis < Repreparation Analysis

Notes:
LA - Libby amphibole
-- = result not available
L = liter
MFL - million fibers per liter
% = percent
CI = confidence interval
TEM = transmission electron microscopy

Repreparation analysis confirms suspected filter mix-up at the laboratory during the original analysis.

TABLE 2
LIBBY OU3: PHASE V PART B, 2013 EYED EGG STUDY, WATER SAMPLING RESULTS
REPREPARATION RESULT COMPARISON

Repreparation Type	Media Type	Index ID	Original Analysis (EMSL-Libby)			Repreparation Analysis (EMSL - Cinnaminson)			Poisson Rate Comparison (90% CI)
			Total LA Structures	Sensitivity (1/L)	Total LA Conc (MFL)	Total LA Structures	Sensitivity (1/L)	Total LA Conc (MFL)	
Reprep from filter	Surface Water	P5-20290	27	1.4E+06	**38**	25	9.8E+05	**25**	[0.96-2.57] The rates are not different
	Surface Water	P5-20294	27	1.2E+06	**34**	25	8.9E+05	**22**	[0.92-2.48] The rates are not different
	Pore Water	P5-20299	28	2.5E+06	**70**	50	2.5E+06	**123**	[0.37-0.86] Rate 1 is less than Rate 2
	Surface Water	P5-20300	0	1.2E+05	**0**	6	1.3E+05	**0.8**	[0-0.59] Rate 1 is less than Rate 2
	Surface Water	P5-20309	26	1.3E+06	**35**	25	2.5E+06	**61**	[0.34-0.93] Rate 1 is less than Rate 2
	Pore Water	P5-20336	1	8.3E+04	**0.08**	2	8.6E+04	**0.2**	[0.02-6.16] The rates are not different
	Pore Water	P5-20324	27	1.3E+06	**36**	37	1.3E+06	**48**	[0.47-1.16] The rates are not different
	Surface Water	**P5-20325**	**26**	**1.7E+06**	**43**	**25**	**1.1E+05**	**2.6**	**[9.93-27.01] Rate 1 is greater than Rate 2**
	Pore Water	**P5-20326**	**0**	**7.8E+04**	**0**	**33**	**1.6E+06**	**54**	**[0-0] Rate 1 is less than Rate 2**
						46	**1.6E+06**	**75**	**[0-0] Rate 1 is less than Rate 2 ****
	Surface Water	P5-20331	25	3.7E+05	**9**	25	6.5E+05	**16**	[0.34-0.94] Rate 1 is less than Rate 2
Reprep from water	Pore Water	P5-20338	34	1.7E+06	**56**	32	1.3E+06	**41**	[0.88-2.11] The rates are not different
	Surface Water	P5-20341	25	2.4E+05	**6**	25	2.5E+05	**6**	[0.57-1.58] The rates are not different
	Pore Water	P5-20348	30	1.7E+06	**50**	32	9.2E+05	**30**	[1.07-2.64] Rate 1 is greater than Rate 2
	Surface Water	P5-20356	25	7.1E+05	**18**	31	1.1E+06	**33**	[0.33-0.86] Rate 1 is less than Rate 2
	Pore Water	P5-20352	0	8.3E+04	**0**	3	8.5E+04	**0.3**	[0-1.67] The rates are not different
	Pore Water	P5-20363	0	1.3E+05	**0**	0	1.3E+05	**0**	Both counts are 0; the rates are not different
	Surface Water	P5-20369	0	1.2E+05	**0**	0	1.3E+05	**0**	Both counts are 0; the rates are not different

All filters pass the CHISQ test for filter loading evenness.

Original Analysis > Repreparation Analysis
Original Analysis < Repreparation Analysis

Notes:
LA - Libby amphibole
-- = result not available
L = liter
MFL - million fibers per liter
% = percent
CI = confidence interval
TEM = transmission electron microscopy

These samples were selected for repreparation analysis by EMSL-Cinnaminson because it was suspected that the paired pore water and surface water results were mixed up by EMSL-Libby. The filter repreparation results confirm that the results were reported incorrectly by EMSL-Libby.

**EMSL-Cinnaminson performed a second repreparation for this filter which confirmed the first repreparation.